A Novel MicroElectroMechanical System (MEMS) Device for Passive Sampling of Hydrophobic Compounds

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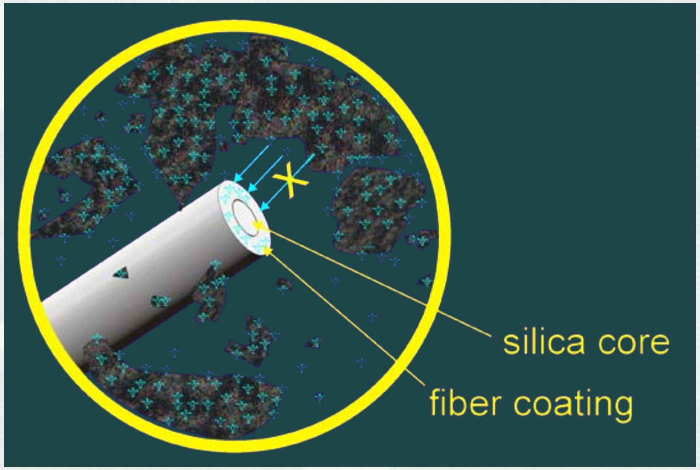
Problem

- Contaminant assessment (chemical analysis) for emergency response, clean up, and NRDAR has significant room for improvement
  - Current approaches are costly
  - Need for measures of bioavailability
  - Spatial and temporal challenges
- Few technological improvements in chemical analysis since NEPA in 1969
  - Example: Using approaches developed in 1970's, it is estimated the Deepwater Horizon spill cost around $20 million to measure "non-detects."

It is Time to Advance our Technology!

Technologies: Passive Samplers

- Passive samplers can be placed *in situ* to sorb contaminants; provide information about bioavailability

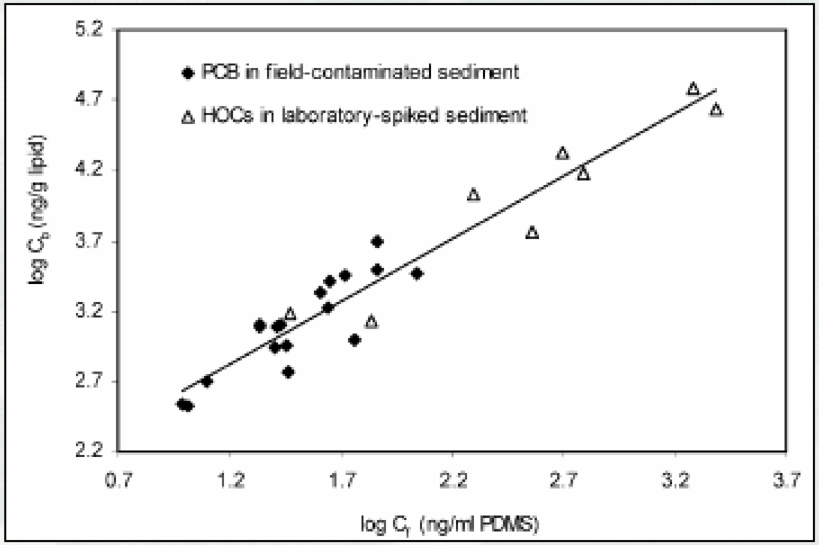


SPME fiber

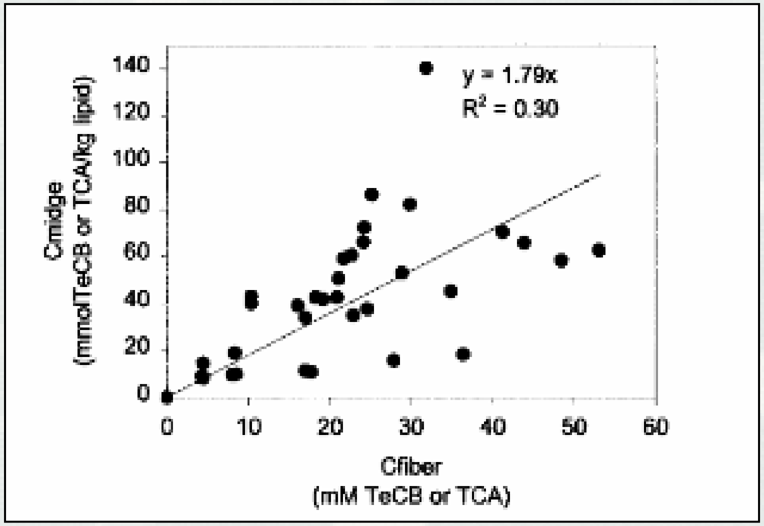
ESTCP project, Reible and Lotufo

- Samplers are removed, extracted for CoC, analyzed
  - For organics: solid phase micro extraction (SPME) fibers, semi-permeable membrane devices (SPMD), polyoxymethylene (POM)
  - For metals: diffuse gradients in thin films (DGT)

Applications to Predict Bioaccumulation



SPME concentrations were predictive of tissue concentrations of PCBs in field-contaminated sediments and laboratory-spiked sediments
You et al. 2006, EST, 40: 6348

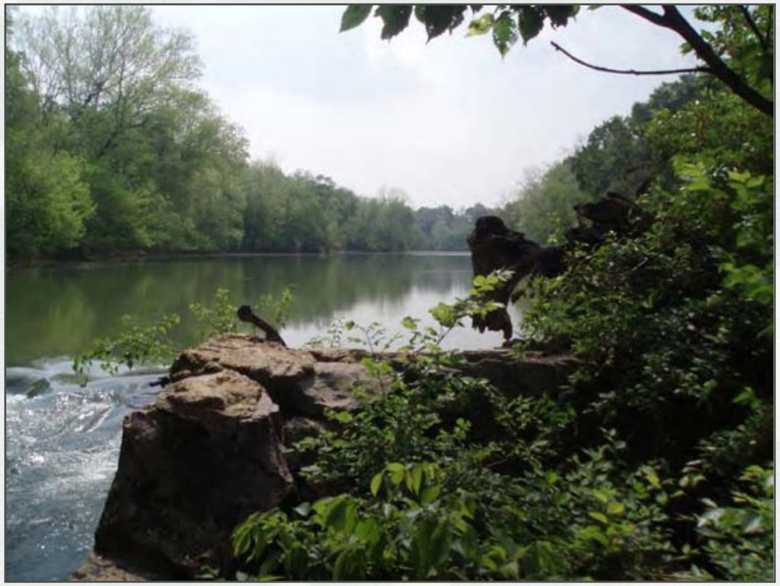


SPME concentrations were predictive of tissue concentrations of chlorinated hydrocarbons
Leslie et al. 2002, ETC, 21:229

SPME fibers can be used to predict bioaccumulation

Application in Risk Assessment

- Anniston Alabama Site
- Using passive samplers to assess bioavailability of PCB and confirm bioassay results



http://www.youtube.com/watch?v=ZZJtxmN9hX8

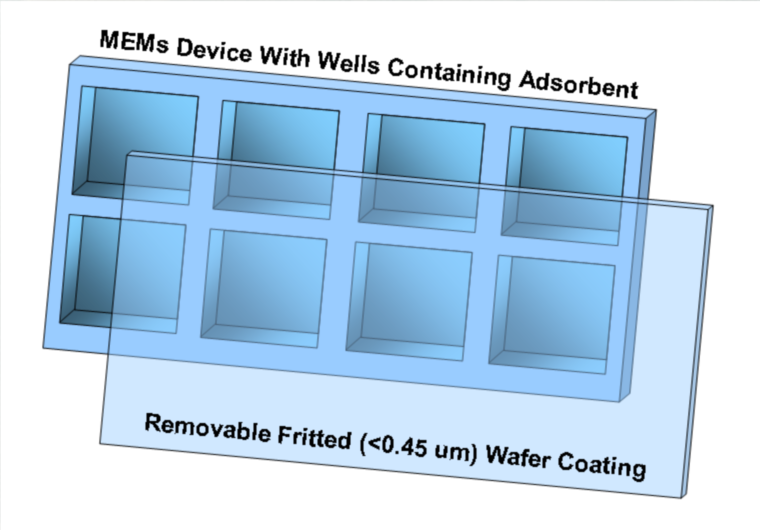
Technologies: Passive Samplers

- Uses:
  1. Measure bioavailability of CoC; direct measure of bioavailable fraction
  2. Use as a line of evidence (LOE) with in a weight of evidence approach
- Benefits: relatively easy and inexpensive; majority of cost is from chemical analysis
- Limitations: fragile, fouling, problems detecting compound on a small fiber

SPME fibers are an opportunistic technology; can we design a technology that intended for sampling contaminants?

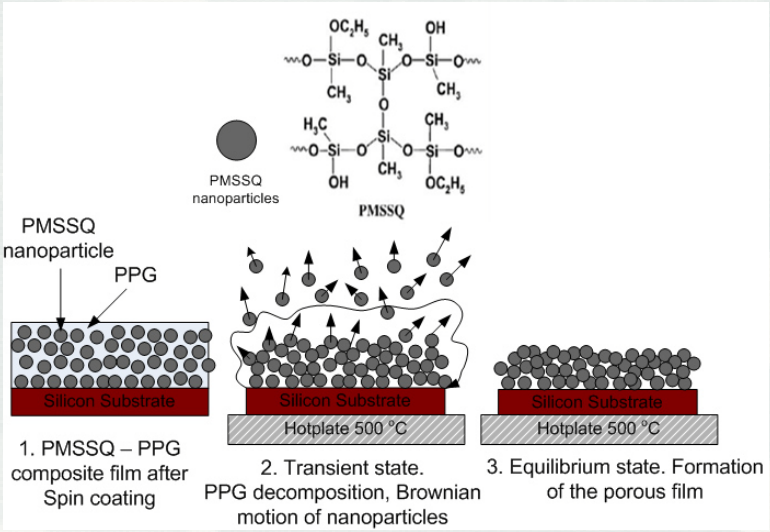
Vision

- Develop a relatively inexpensive sampling device for a wide range of contaminants
- Could be used to sample or develop a detection system
- Immediate needs:
  1. Develop a sorbant surface with a high affinity for contaminants
  2. Robust and stable in environmental conditions
- Next steps:
  1. Detection
  2. Reporting



Sorbant Surface Material

- Sampler surface was fabricated using organosilicate nanoparticles ~ 3nm in size as the building blocks
- OSNP applied on a silicon substrate at different temperatures
  - 250-550°C



Polymethylsilsesquioxane (PMSSQ, ~ 3nm size), dispersed within polypropylene glycol (PPG).

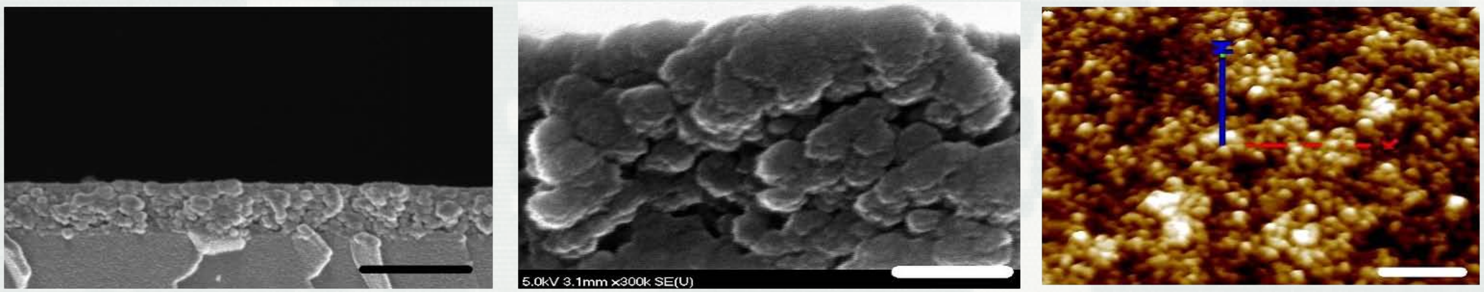
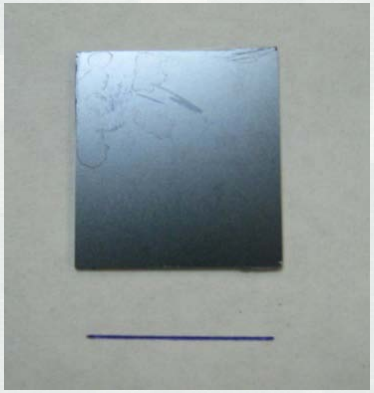


Figure 3 a),b)Cross sectional SEM image of the NPO film c) AFM image of the NPO film surface

Cross section (SEM) and surface of OSNP (AFM)

The Sorbant Surface

- Surfaces applied to a silicon chip and characterized
  - 1 cm^2
  - Around 1.5 um thick



Sample	Thickness
NPO-5555-250°C-5min	1689 nm
NPO-5555-350°C-5min	1535 nm
NPO-5555-450°C-5min	1439 nm

Image of NPO film and ellipsometry results

SPME

- Length: 2.5 cm
- Diameter: 230 um
- Surface area: <u>18 mm^2</u>

OSNP Surface

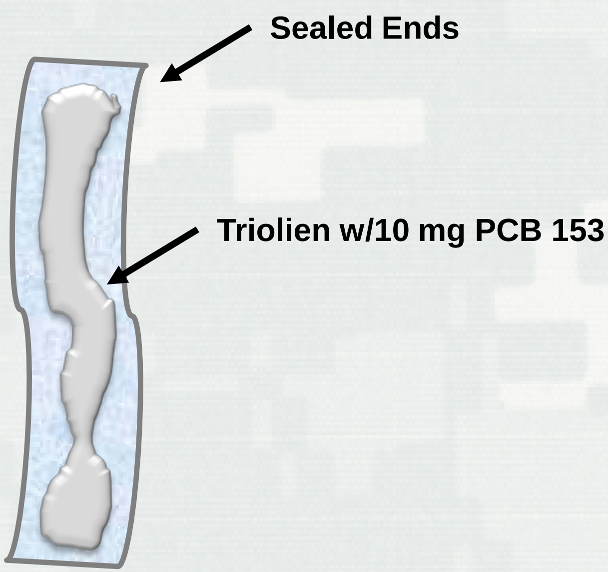
- Length: 1 x 1 cm
- Thickness: 1.5 um
- Surface area: <u>1800 mm^2</u>

100X increase in sorption surface area!

Testing the Surfaces

Goal: Compare the sorbtion of SPME versus OSNP Chips

- Step 1: prepare a test media (PCB153)
  - PCB 153 in water
  - Concentration using a passive “dosing” system
  - SPMD tube with 0.5 g of glyceryl trioleate (triolien) + 10 mg PCB
  - Achieves water concentration of around 0.0059 $\pm$ 0.002 ng/ml

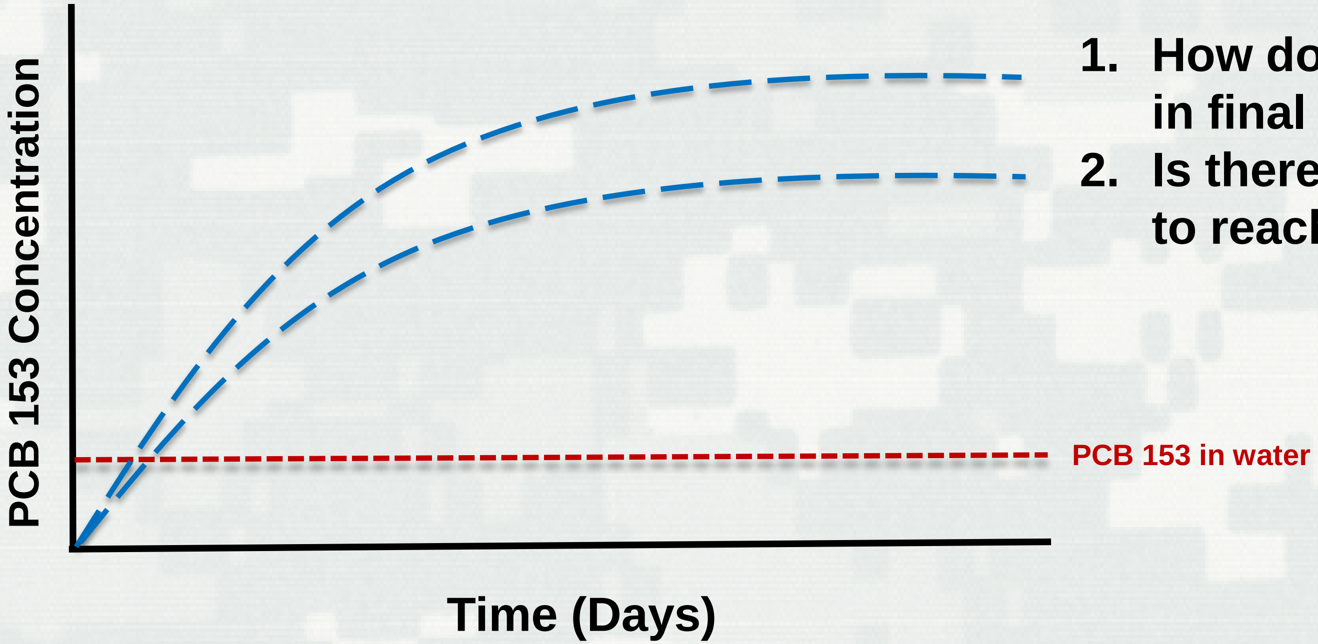


SPMD

SPMD with PCB in water and aeration

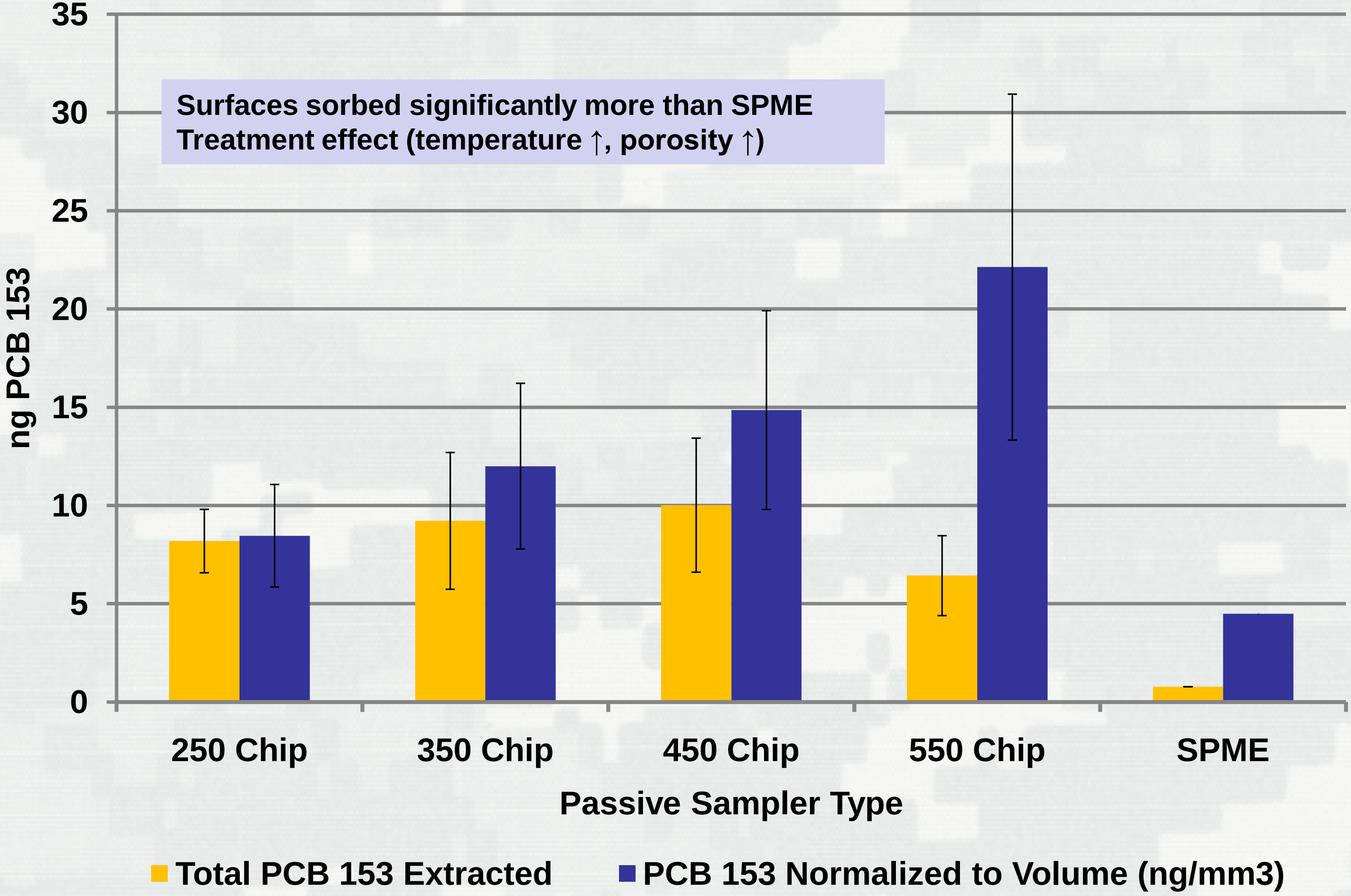
Testing the Surfaces

- Exposure to surfaces
  - Allow SPME and surfaces to equilbrate with water; 7 days with PCB 153 in water
  - Remove and extract through procedure to dewater (methanol) then hexane
  - Analyzed by GC-MS



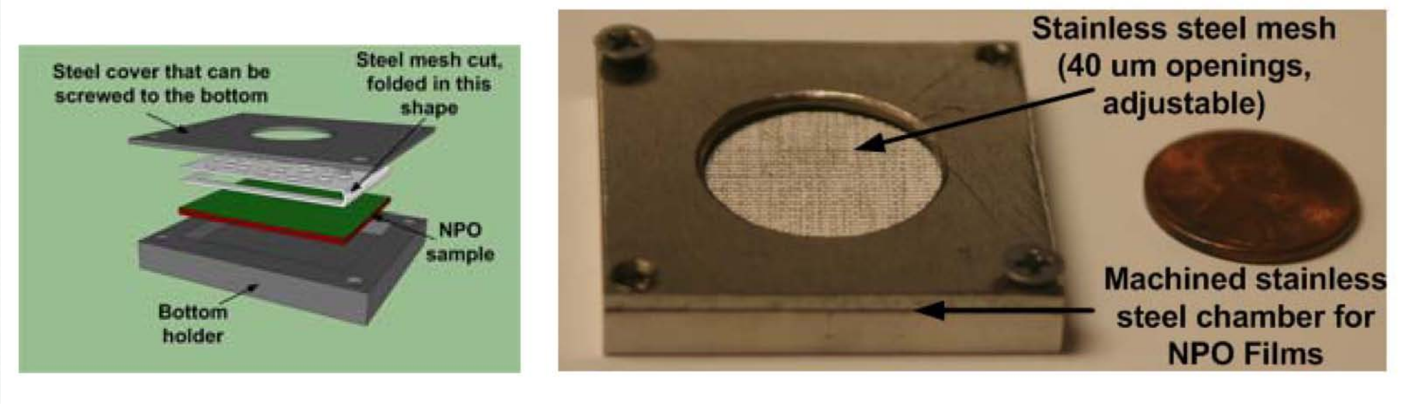
1. **How do surfaces compare in final concentration?**
2. **Is there a difference in time to reach equilibrium?**

Results: Sorption of PCB 153 on Samplers

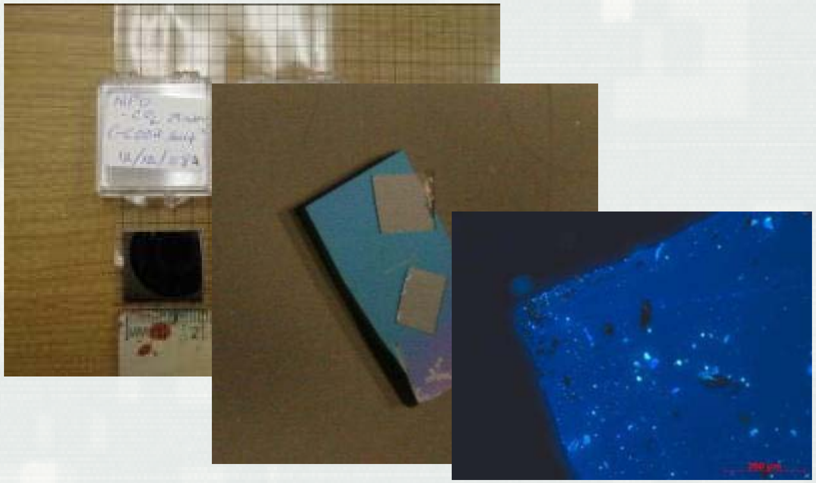


Field Deployable Device

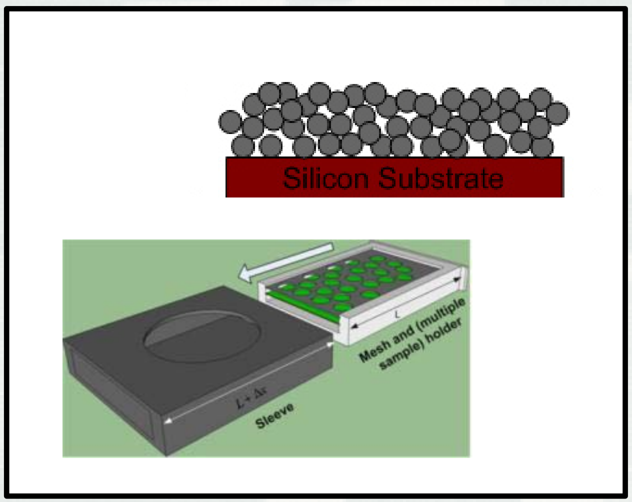
- Developed and testing a field deployable device
  - OSNP surface in a steel chamber with screen
  - Current device is 100% teflon with silica OSNP surface
- Being tested at Anniston Site to compare to SPME data; for the purpose of supporting bioaccumulation and toxicity assessment



Conclusions



Nano Porous Organosilicate (NPO) Films



- Develop samplers for *in situ* analysis of CoCs
- Technology
  - Initial development focused on sorbent materials with increased surface area
  - Deployable devices that are robust and recoverable
- Future research focuses on integrated detection methods within a MEMs platform
- Help: Always need help with field test sites

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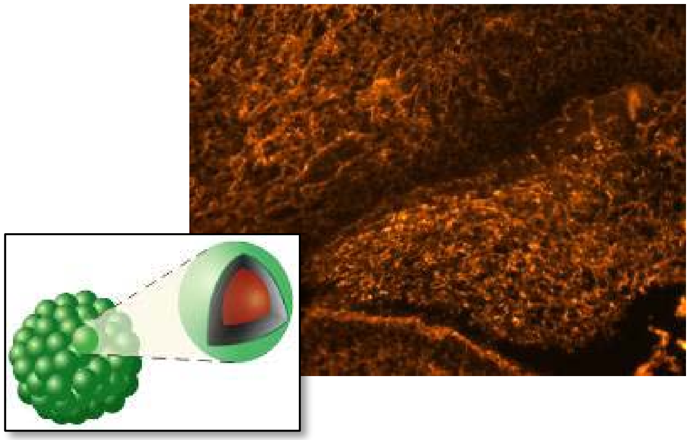

Labeled OSNP in Chicken Heart, Bok et al., 2010